

## LEATHER LUBRICANT RESISTANT TO DRY-CLEANING SOLVENTS\*

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### ABSTRACT

This report describes the use of a long-chain amino acid ( $\text{RNHCH}_2\text{-CH}_2\text{COOH}$ ) as a leather lubricant that is resistant to removal from the leather by common dry-cleaning agents. Data are presented to indicate the relatively low removal by Stoddard solvent, perchloroethylene, and 1,1,2-trichloro-1,2,2-trifluoroethane. Similar test data on conventional leather lubricants showed these to be lost to the extent of two to four times as much. The dry-cleaning tests were run on 10 x 10 inch pieces of suede leather in commercial equipment.



### INTRODUCTION

A survey of recent literature (1-5) concerned with the dry cleaning of leather has indicated that retention of fatliquor (fiber lubricant) is still an important problem. There seems to be good agreement that oil put into the leather by the tanner is removed by the dry-cleaning solvent to such an extent that the cleaning process tends to make the leather harsh (1, 3). As a result, practically everyone recommends that the dry-cleaned garments be "re-oiled" after cleaning by rinsing (2) or dipping the garment in an oil-solvent bath (*i.e.*, 20 grams fat/liter of solvent) (5), or by spray treatment with a solution of an oil in an organic solvent. One report cautions that certain fats normally used in leather processing should not be applied by the spray method (2).

The need for lubricants resistant to dry cleaning is obvious. Some work to develop such materials has been published (1, 3). A report by Garrett (1) in 1964 shows experimental data on four leathers treated with two fatliquor mixtures that withstood extraction by mineral spirits or perchloroethylene considerably better than general purpose fatliquors.

One of these fatliquors was a fully sulfited oil, with anionic emulsifiers, which gave reasonable results in all four leathers but was best on full chrome suede.

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The other was a mixture of 60 parts sulfited oil (fattening substance 80 percent) ; 20 parts sulfated fatty acid tanning agent (50 percent active substance) ; 20 parts complex fatty alcohol and non-ionic emulsifiers. This mixture gave the best results in the test leathers. The data indicate that 75 to 95 percent of the fattening substance is bound to the hide fiber and chrome complex, since it resisted extraction by the cold solvent or by solvent dry cleaning. The report states also that the mixture of sulfited oil, sulfated fatty acid tanning agent, and complex fatty alcohol gave excellent softening properties.

Our paper is concerned with a study of the use of certain fatty amino acid derivatives as leather lubricating agents, and of their behavior when subjected to various dry-cleaning agents. The compound used for these tests is a member of a group of amphoteric surfactants developed for the cosmetic industry about ten years ago (6-9). Many other uses (9) have been developed since these compounds were introduced.

These fatty substances are members of the series represented by the general formula:



where R is a long-chain hydrocarbon radical and M is hydrogen or a salt-forming cation. Various amphoteric materials of this particular type are currently available under the name "Deriphat."‡ These fatty amino acid derivatives are obtained by the interaction of a fatty primary amine and methyl acrylate. The aminopropionic ester thus formed is then hydrolyzed to the carboxylic salt or acid (6). In the case of Deriphat 151C, the supplier's descriptive formula is: *N*-"coco"- $\beta$ -aminopropionic acid, indicating that the long-chain hydrocarbon radical in structure (I) above was derived from coconut oil and is a mixture of compounds. A more refined form is listed as Deriphat 170C, with a somewhat more definite chemical description indicating this to be a mixture of *N*-lauryl- and *N*-myristyl- $\beta$ -aminopropionic acids.

The chemistry and physical properties of these compounds are discussed in detail in References 6-10. Their versatility is derived from their internal electron properties, their multiple-function surfactant-detergent capabilities in one compound, and their substantivity.

## EXPERIMENTAL

A complete description of the skins, tannages, lubrication with Deriphat 151C, and the dry-cleaning procedure follows:

### Skins

The New Zealand lambskins used in this study were obtained from a garment suede tanner after they were degreased, chrome tanned, and retanned with a

‡Mention of brand or firm names does not constitute an endorsement by the Department of Agriculture over others of a similar nature not mentioned.

vegetable tanning agent. The tanner's degreasing treatment with petroleum ether lowered the average fat content of the skins to below three percent. The skins were removed from the production line by the tanner just prior to the fatliquoring step and sent to our laboratory.

### **Retannage with Glutaraldehyde (12, 13)**

Several of the skins described above were retanned for 6.5 hours using 25 percent commercial glutaraldehyde in the amount of ten percent of the wrung weight of the skins, in a 100 percent float, at pH 3.9–4.0, and at the temperature range of 42 to 53°C. The skins were then washed in running water to remove unused glutaraldehyde, wrung, and refrigerated to await further processing. The shrinkage temperature of this leather was 116°C. (in water under pressure (14)), essentially the same as that for similar skins that were not retanned.

### **Fatliquoring**

Several skins that were retanned with glutaraldehyde were returned to the co-operating tannery, along with several that were not retanned, to be treated with the fatliquors they normally use for garment suede and processed into finished leather.

### **Lubricating with Deriphat**

Several groups of skins, some retanned with glutaraldehyde and some not retanned, were treated in our laboratory with Deriphat 151C as the lubricant.

This was done in a drum with the following solution: warm water, 50 percent of the drained weight of the skins; Deriphat 151C, 12 percent (same basis); Dow-Corning Reagent B (anti-foam), several drops. The drum was warmed with heat lamps which kept the temperature of the float at 46–50°C. Within 30 minutes of drumming, the skins absorbed most of the float. The pH was 5.1.

In several runs, the pH of the float was adjusted to 4.0 by adding the required amount of five percent acetic acid solution and running the drum for an additional 15 minutes.

All the skins treated with Deriphat 151C were hand squeezed when removed from the drum, placed in polyethylene bags, and stored in a refrigerator for a short time, pending delivery to the tannery. There they were dried and processed (with no additional fatliquor) into suede garment leather.

All the leather returned by the tanner was quite normal. Several of the skins treated with Deriphat were a little softer than most of the others. The nap on all was good. The change in shrink temperature was slight; the lubrication with conventional fatliquor or Deriphat dropped that value about 5°C.

### **Sampling Leather for Dry-Cleaning Tests**

A set of four pieces, approximately 10 x 10 inches each, was cut from all ex-

perimental skins for dry-cleaning tests. These pieces were cut from the center part of the skin; the flanks, neck, and tail portions were discarded. A three-inch strip of leather from between the upper two and lower two test pieces was cut into four parts (1.5 x 10 inches), and each part was analyzed separately for oil content (ALCA chloroform extractables) (11). This analysis gave a value for a location near each test piece before dry cleaning, and was considered necessary to overcome, in part, the effects of variation in oil content of leather from place to place in each skin. After dry cleaning the 10 x 10 inch test pieces, a 1.5 inch strip was cut off each from the edge that had been adjacent to the strip analyzed previously, and the residual oil content was determined.

### **Dry-Cleaning Tests**

Six sets of leather as described above, one set from each tannage-fatliquor combination and from commercial suede, as listed in Table I, were sent out for dry cleaning under standardized schedules as described below. The dry-cleaning schedule was arranged so that each set was cleaned apart from any other set in order that there could be no transfer of material by the cleaning solvent from one set of leather to another. The cleaning cycles were repeated so that in each set of four pieces, one piece was cleaned once, a second piece twice, the third piece three times, and the last piece four times. The dry-cleaned pieces were returned to this laboratory for analysis.

### **Dry Cleaning with Valclene**

The dry cleaning was done at the Dupont Dry Cleaning Products Laboratory (Electrochemicals Dept.) in a Vic Model 141 Dry Cleaner, using their regular procedure — a 3.5 minute wash followed by a two minute detergent-free rinse. The leather pieces were then extracted (centrifuged) for 2.5 minutes, followed by a five minute drying period. This procedure was repeated for each of the four cycles mentioned above.

### **Dry Cleaning with Stoddard Solvent**

The dry cleaning was done at the laboratory of the National Institute of Dry Cleaning. The work was done in a commercial machine of thirty-five pound dry weight capacity. Stoddard solvent was used, with no detergents. The procedure was 20 minutes agitation in solvent, followed by three minutes in a centrifugal extractor. The samples were cabinet-dried at about 130°F. The leather samples were pinned to garments normally used for the purpose of bringing the load up to 35 lbs. The dry-cleaned pieces were returned to this laboratory for analysis.

### **Dry Cleaning with Perchloroethylene**

The dry cleaning with perchloroethylene was also done at the National Institute of Dry Cleaning Laboratory. The leather samples were pinned to one garment in an eight-pound dummy load and were in contact with solvent for eight

minutes. This was followed by a three to four minute extraction. Finally, the leather pieces were removed and hung to air-dry at room temperature. The dry-cleaned pieces were returned to this laboratory for analysis.

#### DISCUSSION AND CONCLUSION

Substantivity is the adsorption of a surfactant at a solid surface from a liquid. The Deriphats demonstrate this property by the affinity of the fatty amino acid molecule for skin, hair, etc. It has been proposed that this affinity is due to the similarity in amphoteric properties of the substrate and the fatty amino acid (6, 9, 10). The substantive properties of most ampholytes appear most effective under neutral or somewhat acidic conditions (9), and are probably an important factor contributing to the resistance of Deriphath to removal by dry-cleaning solvents.

The compounds, when used as lubricants, can exhibit anionic or cationic characteristics, depending on whether the system is alkaline or acid. An equilibrium of all three forms exists at all except the most extreme conditions of acidity or basicity (6, 9, 10):



Acid Range  
(cationic)

Isoelectric Range  
(neutral)

Alkaline Range  
(anionic)

M = hydrogen or salt-forming cation

Deriphath 151C was selected from a group of six related compounds, and was used as a lubricant, alone. Since this compound is soluble in water, it was applied as a water solution. This method of application is an aqueous impregnation rather than conventional fatliquoring, in which oils are exhausted from an emulsion. Uptake of lubricant was more difficult to evaluate and was estimated from a determination of fat content of the spent Deriphath solution. With a fairly short float (50 percent on weight of drained skins), the pickup of Deriphath averaged 86 percent of the quantity applied. This was determined by evaporating to dryness samples of liquid squeezed from the skins at the end of the treatment, and comparing this value with the amount of Deriphath offered, taking into account all the liquid in the system, including water in the tanned skin before lubricating.

Our primary interest in Deriphath 151C was aroused by its relatively low solubility in organic solvents, particularly carbon tetrachloride, perchloroethylene, and mineral spirits, and the consequent potential of its use as a lubricant resistant to removal by dry-cleaning solvents. Although the supplier reports solubility of the 50 percent aqueous solution of Deriphath 170C as less than one percent in carbon tetrachloride or in mineral spirits, we found that dry, solid Deriphath 151C was much more soluble in carbon tetrachloride. The solubility of dry

**TABLE I**  
EFFECT OF DRY CLEANING ON CHLOROFORM EXTRACTABLES  
IN EXPERIMENTAL AND COMMERCIAL SUEDE LEATHER

Leather Tannage	Lubricant	Dry- Clean- ing Cycles	Chloroform Extractables,* Before and After Dry Cleaning With:						Perchloroethylene		
			Valienet†			Stoddard Solvent‡					
			Before %	After %	Loss %	Before %	After %	Loss %	Before %	After %	Loss %
Commercial chrome- tanned, vegetable- retanned	Deriphat 151C** (final pH adjusted to 4)	1	7.0	5.7	19.2	6.3	5.7	9.7	6.8	5.5	19.3
		2	7.1	5.7	19.9	6.2	5.8	7.2	6.8	5.1	24.1
		3	6.6	5.5	17.4	6.3	5.9	6.2	6.5	5.2	19.9
		4	7.3	5.6	23.8	6.4	6.0	6.0	6.9	5.5	20.3
Commercial chrome- vegetable-tanned, glutaraldehyde- retanned	Deriphat 151C** (final pH adjusted to 4)	1	7.2	5.8	19.8	6.4	5.0	20.6	7.4	5.8	20.9
		2	6.2	5.1	17.4	6.6	5.0	23.9	7.5	5.9	21.5
		3	6.3	5.3	16.9	6.2	5.1	17.7	6.7	5.6	15.8
		4	6.8	5.8	15.9	6.5	5.6	13.0	8.1	5.9	26.6
Commercial chrome- vegetable-tanned, glutaraldehyde- retanned	Commercial††	1	13.1	4.7	64.4	13.0	4.8	62.9	13.2	4.6	65.3
		2	13.0	4.6	65.0	12.9	4.6	64.5	12.8	4.4	65.9
		3	13.1	4.6	64.9	13.1	4.6	65.0	12.9	4.2	67.4
		4	12.9	4.6	64.5	12.7	4.9	61.5	12.7	4.4	65.5
Commercial chrome- vegetable-retanned	Commercial††	1	10.6	4.0	62.1	10.8	3.5	67.9	11.2	3.9	65.4
		2	10.7	3.8	64.3	10.2	3.2	68.5	11.4	3.8	66.7
		3	11.1	4.2	62.7	10.7	3.6	66.5	11.4	3.5	69.6
		4	10.9	4.3	60.4	10.0	3.6	63.8	11.1	3.7	67.1
Commercial suede	Commercial‡‡	1	11.2	3.6	68.3	12.4	2.9	77.0	12.8	3.3	74.6
		2	10.8	3.7	65.8	11.8	2.5	78.7	12.9	3.2	75.3
		3	11.0	3.8	65.2	12.4	2.7	78.5	12.5	3.2	74.3
		4	10.6	4.3	59.7	12.4	2.6	79.1	12.8	3.2	75.3
Commercial suede	Commercial***	1	7.8	4.4	43.5	8.2	3.5	57.4	7.4	2.9	61.1
		2	8.3	4.1	50.4	7.9	3.2	60.1	7.1	2.6	64.0
		3	7.9	4.0	50.1	7.7	3.5	55.4	7.3	2.8	61.9
		4	8.2	4.1	50.7	7.8	3.5	55.2	7.3	2.7	62.4

\*ALCA Provisional Method B4 (1957), moisture-free basis; all determinations run in duplicate.  
†DuPont's commercial dry-cleaning agent (1,1,2-trichloro-1,2,2-trifluoroethane).  
‡Petroleum base solvent specially refined for dry cleaning; specifications by U. S. Department of Commerce; white spirits.  
\*\*\*One of General Mills' amphoteric surfactants (N-"cocoyl"-β-aminopropionate).  
††Fatlignor normally used for garment suede by Tanner #1.  
‡‡Fatlignor normally used for garment suede by Tanner #2.  
\*\*\*Fatlignor normally used for garment suede by Tanner #3.

Deriphat 151C in Stoddard solvent (mineral spirits) or "Valclene" is quite low. At room temperature, saturated solutions of Deriphat 151C in the following solvents were found to contain the following percentages of Deriphat 151C: chloroform, more than 20; carbon tetrachloride, 12.2; perchloroethylene, 11.0; Stoddard solvent (mineral spirits), 2.2; and 1,1,2-trichloro-1,2,2-trifluoroethane, 1.5. The appreciable solubility of Deriphat 151C in chloroform made it easy to analyze for "fat" content by extraction of ground leather samples, as specified by the standard method of analysis (11).

A comparison of the effect of the three dry-cleaning solvents on the removal of lubricating oils (chloroform extractables) from experimental and commercial suede leathers is presented in Table I. The results show that when Deriphat 151C was used as the lubricating agent, less than 27 percent was extracted during four cycles of dry cleaning. Also, it appears that the bulk of the material was extracted during the first cycle. There was not much difference in the amount extracted by the three solvents, except for the case of Stoddard solvent on chrome-tanned, vegetable-retanned suede. In this instance, less than ten percent was extracted. Surprisingly, when the leather was retanned with glutaraldehyde, the amount of lubricant removed by Stoddard solvent was similar to that removed by the other solvents. In either case, the amount of lubricant remaining in the leather ranged between five and six percent, an amount which seems to be sufficient to give softness and a good hand.

On the other hand, leathers lubricated with commercial fatliquors lost between 50 and 75 percent of their oil content during dry cleaning. Again, most of the oil was removed in the first cycle. The effect of retannage with glutaraldehyde on the retention of oil was so slight that it could possibly be within the range of skin-to-skin variation. The glutaraldehyde-retanned leathers did contain slightly higher residual oil after dry cleaning with Stoddard solvent and perchloroethylene. In fact, the amount remaining was comparable to the amount left by Valclene. In most cases, the amount of oil remaining in the leather was less than four percent, an amount which is undoubtedly not sufficient to maintain suppleness and good hand.

Since acidification sometimes caused precipitation of the Deriphat because of a "wide" isoelectric point near pH 4, a separate experiment was made in which the treated leather was left at the ambient pH of 5.1. The resulting suede leathers (and commercial suede for comparison) were subjected to dry cleaning with Valclene and Stoddard solvent. Since the previous test indicated that most of the lubricant is removed in the first cycle, this test was limited to only two cycles. The results in Table II show again that the Deriphat was more resistant to removal by dry-cleaning solvents than commercial fatliquor oils. The higher initial oil content of the experimental leathers resulted from the fact that more Deriphat 151C was picked up by the leather in this experiment than in the previous one.

TABLE II  
EFFECT OF DRY CLEANING ON CHLOROFORM EXTRACTABLES  
IN COMMERCIAL AND EXPERIMENTAL SUEDE LEATHER

Leather	Dry-Clean- ing Cycles	Chloroform Extractables*				
		Before Dry Cleaning %	After Dry Cleaning With:			
			Valclene†		Stoddard Solvent**	
			After %	Loss %	After %	Loss %
Exp. Suede† #1	1	9.17	7.01	23.56	6.07	33.81
	2	9.17	7.46	18.65	5.76	37.19
Exp. Suede† #2	1	8.29	6.68	19.42	5.54	33.17
	2	8.29	6.36	23.28	4.83	41.74
Exp. Suede† #3	1	8.46	6.69	20.92	5.62	33.56
	2	8.46	6.97	17.61	5.57	34.16
Commercial Suede	1	9.82	3.69	62.42	3.25	66.90
	2	9.82	3.55	63.85	3.18	67.62

\*ALCA Provisional Method B4 (1957). See Reference 11. All determinations run in duplicate on a moisture-free basis.

†Leather samples 1, 2, and 3 are commercial chrome-vegetable suede, glutaraldehyde-retanned, lubricated with Deriphat 151C at approximately pH 5.1, not acidified.

‡DuPont's commercial dry-cleaning agent (1,1,2-trichloro-1,2,2-trifluoroethane).

\*\*Petroleum base solvent specially refined for dry cleaning.

But, even at this higher oil content, there was less tendency toward possible surface oiliness than when the oiling solution was acidified.

Although the leathers contained more Deriphat initially, the amount remaining after dry cleaning was only slightly higher than that in the previous experiment. This suggests that some five to six percent of the lubricant is fairly substantive to the leather, and the dry-cleaning solvents removed the excess or less substantive material. It is interesting to note that Deriphat is considerably more substantive to leather under these conditions than are the regular fatliquor oils.

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## DISCUSSION

MR. BATTLES: This paper will be reviewed by Mr. R. N. Jones, A. C. Lawrence Leather Company.

MR. JONES: We are indebted to Mr. Viola and his colleagues for another informative and practical paper. It should stimulate considerable interest among garment suede tanners in particular.

The dry-cleaning problem has been a sore spot with us for many a year. Generally speaking, the consumer has two choices. He can give his garment to the so-called "leather dry-cleaning specialist" and get a reasonably satisfactory job, but pay a high premium for so doing, or he can give it to the "fast service shop on Main Street, U.S.A." and assume a high degree of risk that the results will be something less than satisfactory. Sometimes they're disastrous!

In the case of the commercial leathers, as was shown in Slide #6, we saw that anywhere from 60 to 70 percent of the original fatty material in the leather is removed as a result of dry cleaning. I was particularly interested in these results because they agree very well with the findings obtained a few months ago by a co-operative group of a number of garment suede tanners and 30 so-called leather dry-cleaning specialists.

The results of the latter tests showed that hide substance, combined tanning, insoluble ash, and everything *except* fatty material remained essentially unchanged as a result of dry cleaning. However, the average fatty material loss of these

30 dry cleaners (and, mind you, these were specialists in the field) was 64 percent.

It really drove home the point as to what our problem, or at least the major problem, is with regard to the dry cleanability and the serviceability features of our product.

One special note of interest in connection with these tests was that, in the coin-op equipment in which some of these same leathers were drycleaned, there was only a 26 percent loss of fatty material. This caused one of the dry cleaners in that group, one of the more progressive ones, I might say, to investigate the reasons why this was so. As a result, he has come up with a unique new dry-cleaning process that he has been operating for five months now, which does dry clean garment suede leather without any oil loss.

This new process, of course, is his private property at the moment, and whether or not he will, at any time in the future, license it to be used by other dry cleaners is a matter of conjecture.

And so, while this is a hopeful sign, the ability to merchandise a garment suede leather that can be cleaned by any Tom, Dick, and Harry, in almost any commercial solvent, and without the tender loving care of a specialist is of paramount interest to the suede tanners. This paper shows us the way and perhaps the means to that end.

I would like to ask Sam to kick off a short discussion period by telling us what some of the considerations were that led to the selection of Deriphat 151C as the one to work with. I understand that there are several members of this Deriphat series.

MR. VIOLA: There are six or seven Deriphats, three of which are solid sodium salts. The remaining are in the acid form and are liquids. Another main difference is the carbon chain length. From preliminary experiments, the acid forms seemed more applicable in our work. While Deriphat 151C and 170C are quite similar chemically, we chose 151C because it is considerably lower in cost.

MR. JONES: Do we have a question or two from the floor?

MR. FRANK RUSSELL (Ciba Chemical and Dye Company): The presence of conventional fatliquor materials in leather is well known to contribute a lot to the depth of shade, and the loss of color in dry cleaning is usually the result of the removal of fat rather than of dye.

I would like to know whether there is any indication of the effect of the Deriphats on the depth of shade: whether it intensifies the color or whether it has any other effect on shade.

MR. VIOLA: We didn't see any change in the color. Dick, you can tell him about that — you did the coloring.

MR. JONES: I would confirm that. We saw a number of the skins that were involved in this test work, and I would say that there was very little effect color-wise as a result of the treatment.

MR. S. PANZER (Robson-Lang Leathers, Ltd.): Would you please tell us whether you tried to apply Deriphats only on leathers treated with glutaraldehyde or on other leathers as well, and what was the difference in application?

MR. VIOLA: The application was the same for leathers tanned with or without glutaraldehyde.

VOICE: How was it applied?

MR. VIOLA: We used a 50 percent float in a small experimental drum.

MR. FRANK RUSSELL: That would be a good point to comment upon, if you would: why do you use such a small float?

MR. VIOLA: The main reason for working with a small float is that the skins readily soak up most of the float and this helps to reduce foaming. Also, since the Deriphat treatment is an impregnation from a water solution, a small float allows a more concentrated solution of a given amount of Deriphat.

DR. PRENTISS (Rohm and Haas Company): I noticed, from the data presented on chloroform extractables from your last three slides, that the values for the Deriphat samples before dry cleaning were quite a bit lower than those for the commercial samples.

Is this because less was picked up, or was less extracted because there was an affinity for the hide?

MR. VIOLA: Essentially, the amount shown in the slides under "before dry cleaning" is the amount picked up by the skins plus natural fat and other extractable materials. Assuming the latter two to be constant for all the skins in the group, we found the amounts shown for Deriphat to be ample, compared to the greater amounts shown for standard fatliquors. Values that high for Deriphat would make the skins too soft and soggy. The other column shows that much less was extracted during the dry cleaning.

DR. PRENTISS: So that means the Deriphat samples you evaluated were probably more efficient lubricating agents.

MR. VIOLA: I believe so.

MR. JONES: Thank you again, Sam, for a very fine presentation.

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